

**MACH 5 ELECTROFORMED NICKEL NOZZLE REFURBISHMENT**

**FNAS Investigation of Ultra-Smooth Surfaces**

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## MACH 5 ELECTROFORMED NICKEL NOZZLE REFURBISHMENT

### ABSTRACT

This task is in support of the Quiet Hypersonic Wind Tunnel effort currently in effect at NASA Langley Research Center, VA.

A laminar flow wind tunnel nozzle has been previously fabricated by electroforming pure nickel over a machined and polished two piece stainless steel mandrel. The mandrel was removed leaving the replicate nozzle surface. The nozzle was then pressed into a heavy stainless steel jacket for mounting features and rigidity. The original nickel surface was a replication of the polished mandrel but had degraded due to oxidation. The inside surface requirements are very stringent in order to achieve laminar or quiet flow at the desired pressure and temperature for the specific design of Mach 5. The throat area of the axisymmetric device must have a surface finish with no defects greater than 16 microinches. This requires an RMS average background of about four microinches or better for inspection purposes.

The task objective has been to apply a coating of nickel-phosphorous alloy by catalytic deposition and then polish the inside of the nozzle retaining dimensional and surface finish tolerances as specified per drawings supplied. Since the unit is not an optical component, conventional optical inspection methods for surface finish and figure are not readily achieved. Measurements have been made using surface profilometry.

### SCOPE

The intent of this task was to apply a very hard, fine grain structure metal alloy of nickel-phosphorous. This alloy is much more durable than pure nickel and typically can be polished to a finer finish with less difficulty due to the amorphous nature of the deposit.

Nickel-phosphorous can be deposited very uniformly at about four ten-thousandths of an inch per hour from catalytic processes. This uniformity depends primarily on sufficient mass transport of the heated solution to the part to permit the maximum diffusion limiting nickel alloy deposition rate. Since the part is essentially conical in shape, the surface area varies considerably with a given axial segment. Thus the primary concern with the plating was forcing enough solution through the inside of the part to maintain uniform plating.

Several candidate vendors were surveyed for the plating and the only response was Metal Surfaces, Inc., Bell Gardens, CA. MSI has a good reputation for military standard compliance plat-

ing and had sufficiently large processes in place for the 75 pound nozzle. A suggested process was forwarded but MSI chose not to follow it explicitly, based on standard processes in place and a high assurance of success. The primary concerns were adhesion and pitting of the deposit. MSI stated that it would be impossible to strip the electroless Ni-P alloy from the nickel nozzle. Also, they were only confident that the adhesion would be adequate if internal standard procedures were followed. The electroformed nickel part (Mach 5) was included in the purchase order and was to be used as a test vehicle for the Mach 6 unit which was the desired deliverable item for the original purchase order NAS8 - 38609. This component was plated by MSI prior to plating the Mach 6 unit.

Lapping was to be performed by the Marshall Space Flight Center, Optics Fabrication Branch with assistance from the University of Alabama in Huntsville, Center for Applied Optics.

#### DESCRIPTION

##### Plating:

The plating was specified to be 0.0015 - 0.0020 inches thick. The preferred alloy is high phosphorous of 10% by weight or more to facilitate lapping. This alloy produces a fine grain structure, approaching an amorphous structure. The formation of non-uniform phosphorous distribution is also minimized. Non-uniform alloy deposition can cause diffusion of phosphorous in non-uniform rosette patterns upon heating subsequently affecting the final polishing quality. The alloy plating process was not stated by MCI.

The requested plating process was to clean and then activate the nickel surface for plating by immersion in 25% HCl, 25% H<sub>2</sub>SO<sub>4</sub> and 50% water at 110-120 Deg. F. (complete process included). This will remove the nickel oxide film which forms spontaneously on the nickel. The vendor, however, chose a more heuristic approach of applying a thin film of pure nickel from a dilute nickel chloride - HCl solution which deposits nickel at a very high reduction potential which reduces the oxide and permits an adhesion layer to be deposited in about one or two minutes. Subsequent plating in the electroless Ni-P will be quite adherent.

Unfortunately the "NiCl strike" process required inserting an electrode (anode) inside the nozzle and application of a rather high current density of 50-100 amperes per square foot. Inadvertently and possibly not known to the vendor, the electrode apparently touched the side of the nozzle in at least three places within the narrow region just behind the throat. The reason for this is not clear but may be related to the fact that the nickel is magnetic and with such high current density in a rod it may well have become an electromagnet and as such was at-

tracted to the nickel nozzle. This caused three areas of damage in the form of small pits. When the nozzle was received after plating, it was not evident on the Mach 5 that this damage had occurred. However on the Mach 6 (previously reported) the damage was much more pronounced. However the decision was made to attempt lapping due to the difficulty involved with stripping nickel phosphorous alloy from the pure nickel nozzle. MCI stated they would not be able to strip the part for replating since no known process existed.

A separate lapping fixture was fabricated for the Mach 5 which permitted rotation on a horizontal axis since the unit built for the Mach 6 was too large. The lapping process revealed the pitting almost immediately and it was evident from prior experience that polishing would not remove the serious pits. Nodules had occurred on the sharp lip of the Mach 6 but were not present on the Mach 5. This encouraged the additional time to be spent on the Mach 5 in the hope that the plating was indeed superior to the Mach 6 which had to be reworked.

After the initial lapping effort, it was apparent that the nickel phosphorous alloy must be removed from the pure nickel and replated for both the Mach 6 and the Mach 5 nozzles. This was a serious problem in that the two materials are very similar in chemical dissolution behavior.

Since the plating firm had agreed only to accept the parts on a "Best Effort" commitment it was up to the present investigators to rework the parts using NASA facilities. The Mach 6 was successfully stripped and replated as reported in the final report NAS8 - 38609.

Nickel metal is subject to passivation at high oxidation potential in an oxidizing media. Phosphorous will actually improve this oxidation resistance to corrosion in the general environmental sense. However, by increasing the oxidation to much higher values than would be normally encountered in any environment, a reversal takes effect. The phosphorous alloy will then begin to dissolve while the pure nickel will further passivate. Since the nickel was encased in stainless steel, the nickel would remain slightly anodic when immersed in solution. This is due to the galvanic coupling and the noble nature of the nickel-iron-chromium alloy casing. Tests were performed on nickel plated, nickel-phosphorous over-plated samples. By using a very concentrated nitric acid solution it was possible to remove the alloy coating from the pure nickel deposit on these samples. Above 65% nitric acid (commercial azeotrope) the pure nickel dissolution was less than 0.0001 inch/Hr. Thus although the vendor stated that no process existed for stripping the Ni-P from the electroformed nickel, a satisfactory method was developed.

### Polishing:

The lapping was initially performed in four stages using alpha  $\text{Al}_2\text{O}_3$  from Microgrit, Buehler or Baikowski Industry. The first grit size was 3.0  $\mu\text{m}$ . This was used until a uniform surface was observed. Next the grit was changed to 1.0  $\mu\text{m}$  followed by 0.3  $\mu\text{m}$  and then 0.05  $\mu\text{m}$  for final abrasive polish using the Buehler face pads. A final very fine polish was completed using colloidal silica gel. This removed the fine scratches left by the alumina polishing steps.

This process was performed on the plating as received from Metal Surfaces, Inc. The first plating and polishing attempt failed to produce the needed surface due to plating defects as previously described.

The performance of the previously described Mach 6 nozzle which was stripped and replated was not as good as desired due to low frequency waves residual after the second plating attempt. This unit was then shipped back to NASA MSFC for additional rework. Again serious decisions involved the additional removal of material regarding the thickness and integrity of the final part.

The lapping process was repeated in the large section of the Mach 6 nozzle using modified hones. A portable Taylor-Hobson Surtronic 3-P profilometer was obtained and used to assess the progress of the reduction in amplitude of the waves. The polishing was initiated with a coarser lapping compound of 5 $\mu\text{m}$  alumina and the lapping pads were modified to reduce edge effects. The same sequence of polishing as before was then repeated. After about eight hours of accumulated lapping time with the three and five micron polishing compound the waves were significantly reduced as measured with the small portable profilometer. At this point the amplitude of the waves was measured at the polishing station, to be about 50 microinches peak to valley. Subsequently another 1.5 hours with the five micron and 1.0 with the 3.0 micron polish reduced the short period waves to about 10 microinches and the longer period waves of about 1/2 inch period appeared to be reduced to about 25 microinches. At this time a significant improvement could be seen by observation. However the dull band of plating mentioned earlier was apparently lapped through to the original pure nickel.

Also due to the amount of time of polishing, the front section was not well matched to the re-polished area. When the small area of the throat was lapped to blend the polishing in the larger area, two pits appeared. These were plated with copper as before and subsequently polished with the small hone and also by hand. At this time it appeared evident that the nickel-phosphorous alloy was wearing through to the base nickel at the

same region as the dull streak had originally been. Therefor the polishing media was limited to the 0.05 micron alumina and the colloidal silica gel beyond this observation.

Based on these difficulties in replating and polishing the Mach 6 nozzle the decision was made by Langley personnel that a continued similar effort on the Mach 5 was not warranted since the eventual use of the part was not established.

#### Inspection:

In-process inspection was performed using visual observation only due to the nature of the defects and the decision not to continue.

#### SUMMARY

##### Accomplishments:

The surface of the nozzle has been coated with a nickel-phosphorous alloy of sufficient hardness and corrosion resistance to improve the durability. Due to plating defects which are clearly process related and not inherent, the part was less than the desired quality. Surface finishing processes and lapping media were identified which produced a sub-micron surface finish on the interior plated surface. Defects apparently manifested by the plating were not repaired for the Mach 5 unit as for the Mach 6 due to limited funds and the fact that no use for the Mach 5 unit were identified other than as a test vehicle for the plating of the Mach 6 unit.

##### Recommendations:

In order to absolutely refurbish this very difficult part to the quiet configuration surface perfection, it will be necessary to once again strip and replate the part. Under no circumstance can the plating process requirements be compromised due to lack of available equipment or process validation. This will require a vertical holding fixture and a plating process large enough to hold the part vertically. The process must have sufficient pumps and plumbing by design to continuously supply an abundance of agitation to the interior of the part throughout the process. Filtration must be continuous even during the plating operation.

The chemical activation process used the second time on the Mach 6 unit provided adequate adhesion and the recommendation is that should the Mach 5 part be replated that the "strike" process with an electrode not be used. Note that this is the exact process recommended to the vendor but which he chose not to use. A very accurate temperature controller is required and solution replenishment during the plating process is mandatory unless a very large process is used.

The use of chromium instead of nickel- phosphorous may be considered but tests would need to be performed. In the case of chromium plating, an anode is mandatory and a special design is required to avoid any chance of striking the part and damaging the surface.

Careful machining of lapping heads for the hones will provide a suitable lapping mechanism. A third size intermediate lap set is also required even if it must be designed and built. Nearly all the difficulties encountered to date relate to the attempt to polish plated nickel with inherent defects. This is not to undermine the vendors but to express concern for attempting to process such a large and heavy piece in a process designed for smaller pieces.



**TABLE 1**

**Materials Used:**

**Polishing Pads**

Buehler Chemsmet  
41 Waukegan Road  
Lake Bluff, IL 60044

**Polishing Compounds**

Baikalox  
Alpha Type Premix  
1.0 -.05  $\mu$ m

Baikowski International Corp.  
6006-B Old Pineville Road  
Charlotte, NC 28217

Buehler .05  $\mu$ m

Buehler Chemsmet  
41 Waukegan Road  
Lake Bluff, IL 60044

Polyurethane  
D-65 Shore Hardness  
.030 in. thick  
UNFILLED

James H. Rhodes & Co.  
Route 12-B  
Franklin Springs, NJ 18841

Polyurethane  
D-65 Shore Hardness

Hardman Inc.  
600 Cortlandt Street  
Belleville, NJ 07109

Microgrit 3 $\mu$ m & 5 $\mu$ m  
Untreated

Micro Abrasive Corp.  
Westfield, Mass.

Silica Colloidal  
NALCO 2360

Rodel Corp.  
9495 E. San Salvador Dr.  
Scottsdale, AZ 85258

Webril Wipes  
100% Cotton

Veratec, Inc.  
Graphic Arts Products  
Walpole, MA 02081

# Suggested Plating Process

## THE UNIVERSITY OF ALABAMA IN HUNTSVILLE

CONTRACT TITLE: WIND TUNNEL REFURBISHMENT

AGENCY: NASA MARSHALL SPACE FLIGHT CENTER

DESCRIPTION: ELECTROLESS NICKEL PLATING ON ELECTROFORMED NICKEL

Two electroformed nickel components will be coated with 0.0015 inches of nickel-phosphorous alloy. The deposit quality must support subsequent polishing to near optical quality mirror surfaces. The nickel alloy deposit will be reduced by about 0.00025 - 0.0005 inches thickness when polished to achieve the optical surface required. It is therefore imperative that the nickel deposit be of very high quality to avoid pitting or poor adhesion. The nickel alloy must be at least 10 weight percent phosphorous and may contain up to 2 additional weight percent copper. Chemical etching to improve adhesion is acceptable. No more than 0.0005 inches of the original electroformed nickel surface shall be removed by any etching process. The entire outer structure is 304 stainless steel and should not be plated unless absolutely necessary. Contact NASA for approval if required.

Processes known to produce suitable deposits when properly operated include but are not limited to the following:

Shipley NICULLOY - 22

Shipley DURAPPOSIT - 90

Shipley NIPOSIT - 468

Enthone ENPLATE NI - 425

Enthone ENPLATE NI - 418

McGean ROHCO

M&T Chemicals INC.

The plating is specified to MIL-C-26074D (Feb. 1989). Drawing notes have precedence over the MIL Standard.

The heat treatment is to be  $320 \pm 10$  Deg. F.

The thickness requirement is 0.0015 - .002 inches.

A reference sample will be plated for alloy check and pitting. This alloy sample will be submitted to NASA prior to plating approval for the parts. A bend sample strip per the MIL-C-26074D will be plated and adhesion tested by the supplier.

Vendors must state if they have successful experiences with electroless nickel plating for the purpose of producing polished optical quality surfaces.

Pitting of the electroless nickel deposits may be related to both the process quality and the plating process control. The acceptable plating process will address both by comprising appropriate cleaning and activation steps. One such step is to use a solution of reducing acids for removing trace impurities prior to plating. The original electroformed nickel surface will contain nickel oxide as well as trace impurities. A common solution for the removal of impurities from the surface of electroformed nickel consists of a mixture of hydrochloric and sulfuric acids which require caution for acceptable safety. About 25 Volume percent of each acid (at the standard concentrated value) is required mixed with 50% de-ionized water. The standard concentrated value is 98% for sulfuric acid and about 33% for hydrochloric acid. This solution is used at 120 Degrees F. An alternate process is to use dilute nitric acid (5% by volume of the 67% concentrated acid) at 120 Degrees F.

An adhesion test must be performed by plating a sample of electrodeposited nickel with the electroless process and performing a bend test to determine that no flaking or peeling due to poor adhesion occurs. The sample must be bent 180 degrees over a cylindrical mandrel with a diameter which is 4 times the combined thickness of the substrate and the deposit. A substrate no more than 0.040 inches and no less than 4 times the deposit thickness shall be used. For this task a substrate 0.006 to 0.008 inches thick, plated to 0.0015 - 0.002 inches with the electroless nickel and bent over a mandrel 0.030 - 0.040 inches in diameter would be ideal.

Additional precautions include vendor process control of filtration, agitation and heating as well as chemical analysis and control. The deposition of .0015 - 0.002 inches of electroless nickel-phosphorous represents about 4 hours of plating during which additions will be required to the solution. Automatic controllers are available and assist in the control but are not mandatory.

Parts of high value are typically monitored continuously by the plating personnel. The deposition can be stopped and restarted if trouble occurs for some operations but may leave a striation in the deposit which would manifest as defective lapping later. Therefore the requirements must include uninterrupted plating.

Alloy control is typically specified and may vary within the part. The specification of +/- 1% is acceptable. An alloy average check by chemical analysis of a coupon sample is appropriate and if the overall average is good then the opportunity for the component to be out of specification is low.

Many parts can be stripped and replated if needed and will not be damaged if stripping is not repeated many times. However stripping of electroless nickel-phosphorous from pure nickel will damage the nickel surface. Therefore the parts are not to be stripped and replated unless expressed permission is given by NASA.

Any vendor considered should demonstrate a thorough knowledge of the analytical requirements for his plating process and precision requirements of the final components.

An agreement that visible pitting is unacceptable should be obtained prior to contracting for this work. A thorough knowledge of precision masking and plating is required.

Suggested vendors include:

ACTERON CORPORATION  
851 SHASTA STREET  
REDWOOD, CA 94063  
415/369-5217 415/364-9748 FAX  
ATT. HANS SELLGE

SPEEDRING COMPANY  
PO BOX 5393  
HUNTSVILLE, AL 35814  
205/837-3606 HUNTSVILLE, AL  
205/739-1710 CULLMAN, AL (FAX)  
ATT. JACK McCLANAHAN

MRC INC.  
6455 PARKLAND DRIVE  
SARASOTA, FL 34243  
813/753-8707  
ATT. DAVID HOUSE

METAL SURFACES INC.  
6060 SHULL STREET  
BELL GARDENS, CA 90201-0521  
714/521-4112  
ATT. RICK SCHNECK

D. Engelhaupt